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(71) Applicant(s): Teijin Ltd.
1-11 Minamimoto-cho, Higashi-ku, Osaka-shi

(72) Inventor(s): Akira Shingu
Central Research Lab., Teijin Ltd.
4-3-2 Asahigaoka, Hino-shi

Masao Suzuki
Central Research Lab., Teijin Ltd.
4-3-2 Asahigaoka, Hino-shi

Hitoshi Mikoshiba
Central Research Lab., Teijin Ltd.
4-3-2 Asahigaoka, Hino-shi

(74) Agent: Sumihara Maeda, patent attorney

(54) HEAT-TREATMENT METHOD FOR FILM

CLAIMS

1. A heat-treatment method for film characterized by the fact that the film is rolled out from a roll of said film retained [held] at a retaining temperature lower than the heat-treatment temperature and is guided to a heat-treatment furnace retained at the heat-treatment temperature; with the film being heated almost to the heat-treatment temperature in said heat-treatment furnace; and during that period with spacers being lapped [overlapped] at least at two ends of the film, and with the film heated in said heat-treatment furnace being rolled up continuously onto a roll via said spacers; then with the roll formed via said spacers being retained at the heat-treatment temperature for a prescribed period of time to be heat-treated.

2. The heat-treatment method for film described in Claim 1, characterized by the fact that said retaining temperature is lower than the glass transition temperature of the film to be treated.

3. The heat-treatment method for film described in Claim 1 or 2, characterized by the fact that the roll of said film to be treated is kept in a preheating furnace controlled at the retaining temperature.

4. The heat-treatment method for film described in Claim 1, 2, or 3, characterized by the fact that said spacers are lapped on the film before the film is fed into the heat-treatment furnace.

5. The heat-treatment method for film described in Claim 1, 2, 3, or 4, characterized by the fact that the roll formed via the spacers is kept at the heat-treatment temperature for a prescribed period of time while being rotated in the rolling direction to be heat-treated.

6. The heat-treatment method for film described in Claim 1, 2, 3, 4, or 5, characterized by the fact that said film is a functional film formed by laminating a thin functional film on a plastic film.

DETAILED EXPLANATION OF THE INVENTION

Industrial application field

The present invention pertains to a heat-treatment method for film, which performs a heat-treatment on a long film to obtain a film with excellent thermal dimensional stability. In particular, the method of the present invention can be used to heat-treat plastic products having a laminated photoselective film, transparent electroconductive film, or other functional thin film used in the optical and electrical fields in order to obtain long films with excellent thermal dimensional stability.

Prior art

There are different kinds of film heat-treatment methods, depending on the length of the film. Heat-treatment methods for relatively short films include (1) a method that carries out heat-treatment continuously at a low speed in a tunnel type of heat-treatment furnace, (2) a method that rolls up a film in a relatively loose manner and directly heat-treats the rolled film in a heat-treatment furnace (such as Japanese Kokai Patent Application No. Sho 58[1983]-98219), and (3) a method that heat-treats a roll-shaped film formed by using spacers between rolled layers in a heating furnace (such as Japanese Kokai Patent Application No. Sho 60[1985]-82338). The aforementioned conventional methods, however, have the following problems. Method (1) is an effective method if the heat-treatment time is in the range of several minutes to

tens of minutes. In most cases, however, said time is not long enough. If the heat-treatment time is prolonged, the length of the heat-treatment furnace will also be increased, and the loss during handling of the film will be increased. Also, the manufacturing cost of the heat-treatment furnace will be increased. In method (2), it is difficult to loosely roll up a long film, or there tends to be damage due to uneven rolling. Method (3) overcomes the problems of method (2). However, since an air layer is present between the films, the heat conduction between the film layers becomes poor. As a result, it will take a long time to raise the temperature of the entire roll-shaped film. In other words, there is a temperature distribution in the length direction of the rolled film, so that it will take a long time until the entire film reaches a uniform temperature. As a result, the heat-treatment temperature and time vary significantly between the part where rolling started, the middle part, and the part where rolling ends along the length direction of the film. The thermal dimensional stability of the film also varies along the length direction of the film. In other words, it is difficult to perform a heat-treatment uniformly over the entire film.

Said method (1) is usually used to heat-treat relatively long films if the heat-treatment time is short. However, no useful method has so far been found to treat long films requiring long heat-treatment times. Regardless of the method used, it is necessary to avoid scratches and deformation, blocking, etc., caused by thermal shrinkage of the film.

Objective of the invention

The objective of the present invention is to solve the aforementioned problems of the conventional technology by providing a heat-treatment method for a long film, which can prevent deformation, blocking, scratching, etc., caused by thermal shrinkage of the film, and that can be performed uniformly along the length direction to obtain a good film.

Configuration and effect of the invention

The aforementioned objective is realized by the present invention as follows. That is, the present invention provides a heat-treatment method for film characterized by the following facts: the film is rolled out from a roll of said film retained at a retaining temperature lower than the heat-treatment temperature and is guided to a heat-treatment furnace retained [held] at the heat-treatment temperature; the film is heated almost to the heat-treatment temperature in said heat-treatment furnace; during that period, spacers are lapped at least at two ends of the film, with the film heated in said heat-treatment furnace being rolled up continuously onto a roll via said spacers; the roll formed via said spacers is then retained at the heat-treatment temperature for a prescribed period of time to be heat-treated.

As described above, according to the present invention, since there are spacers between the rolled film layers, the occurrence of blocking, scratches, etc., between the film layers can be

prevented. Also, since the film on the roll-out side is kept at the retaining temperature below the heat-treatment temperature, the influence of heat on the roll in the roll-out part can be eliminated. On the other hand, the discharged film is rolled up via the spacers along the two ends of the film between the roll-up layers while being heated to the heat-treatment temperature. In this way, hot air, at the same temperature as the heat-treatment temperature, can be blown into the space between the film layers. Consequently, the temperature between the rolled film layers becomes uniform, and an ideal uniform heat-treatment state can be achieved along the length direction.

Also, when the film is kept at the heat-treatment temperature after being rolled up, if the roll-up side is rotated continuously, the deformation or blocking, etc., caused by sagging of the film can be prevented, so that a better heat-treatment can be performed.

Although the retaining temperature is preferably high in order to reduce the temperature difference when the film enters the heat-treatment furnace and to shorten the heating time in the heat-treatment furnace, it should not be too high in order to prevent thermal deformation in the roll during rolling. It is usually set to a temperature that virtually does not affect the quality (occurrence of scratching, etc.) of the film during rolling. From this point of view, the retaining temperature is set to a temperature below the glass transition temperature of the film. A preheating furnace that blows hot air can be used as the means for retaining [holding] the film at the retaining temperature. However, said means is not needed if the temperature of the air can be used as the retaining temperature.

The method disclosed in the present invention can generally be used for heat-treatments that require setting at a prescribed heat-treatment temperature for a prescribed period of time or longer, such as the heat-treatment needed for adjusting the thermal characteristics of a plastic film itself, to be described later, or the heat-treatment needed for improving the electrical characteristics, optical characteristics, mechanical properties, heat resistance, humidity resistance, etc., of films obtained by forming a transparent electroconductive film, photoselective film, or other functional films on said plastic films used as a substrate by means of vacuum deposition, sputtering, chemical coating, etc. The method of the present invention is especially desired for a heat-treatment with a long heat-treatment time, for example, for the transparent electroconductive film used in the application example to be described later. Consequently, there is no special limitation on the film that can be used in the present invention as long as it can withstand said heat-treatment. The plastic films or films formed by laminating said functional films on the plastic films used as substrates can be used.

Examples of the film include polyethylene terephthalate film, polyethylene naphthalate film, polystyrene film, polycarbonate film, triacetate film, polysulfone film, polyether sulfone film, polyimide film, polyamide imide film, etc. It is preferable to use a polyethylene

terephthalate film in consideration of the heat resistance, humidity resistance, and thermal dimensional stability.

In the following, the present invention will be explained in more detail with reference to figures.

Figure 1 shows the configuration of the apparatus used in the application example of the present invention. In Figure 1, 1 represents a film roll on the roll-out side, 2 represents a spacer arranged at the two ends of the film, 3 represents a roll formed via spacers 2 as shown in Figure 2, 4a represents a preheating furnace, and 4b represents a heat-treatment furnace. Said preheating furnace 4a and heat-treatment furnace 4b are the well-known hot air furnaces, whose temperatures can be controlled independently. The roll 1a of film 1 that has undergone the prescribed treatment is installed in the roll-out part of preheating chamber 4a, and the roll 2a having spacers 2 arranged at the two ends of the film is installed in the same way. There is no special limitation on said spacers 2, which are arranged at a certain interval. However, it is preferred that the spacers be the same kind [material] as heat-treated film 1 if heat-treated film 1 thermally shrinks. If spacers 2 are the same kind as heat-treated film 1, they will have almost the same thermal shrinkabilty. Even if thermal shrinkage occurs due to the heat-treatment performed after rolling, the deformation caused by uneven rolling or tight rolling can be minimized.

The film 1 fed out from roll 1a on the roll-out side in preheating furnace 4a is overlapped by guiding roll 5 along with spacers 2. In this case, since spacers 2 have contact with guiding roll 5 instead of the film 1 to be heat-treated, said film 1 can be prevented from being scratched by guiding roll 5. Since the temperature of film 1 and spacers 2 entering heat-treatment furnace 4b are raised close to the heat-treatment temperature until the time they rolled up, thermal shrinkage starts, and a certain degree of thermal shrinkage will occur until [the film] is rolled up. A heat-treatment time in the range of 1-10 min is usually long enough.

If spacers 2 have a larger width and thickness, the interval between the film layers can be increased. However, the cutoff length at the end of the film for removing spacers 2 will also be increased. As a result, the loss of the film in the width direction will be increased, and the diameter of film roll 3 will be increased. This is undesired. Consequently, the width of spacers 2 is preferably in the range of 1/10-1/50 of the film width, and the thickness is preferably in the range of 50-500 μm. If film 1 is wide, spacers 2 can also be arranged in the middle besides the two ends of the film. When bumps and dips are formed by performing lining on said spacer 2, a certain degree of ventilation [exposure to air] becomes possible besides the air between the film layers of film roll 3.

It is also possible to perform the conventional pretreatment, such as coating, on said film 1 and separators 2 in advance in order to restrain the generation of oligomers, etc.

The heat-treatment temperature can be selected appropriately corresponding to the purpose of the heat-treatment. If film 1 includes a plastic film, the heat-treatment temperature should not exceed the glass transition point of that plastic film. In the case of a polyethylene terephthalate film, the heat-treatment temperature is usually in the range of 120-180°C. The time needed for the heat-treatment of said film roll 3 varies depending on the purpose. The higher the heat-treatment temperature, the shorter the heat-treatment time, which is usually longer than 30 min. In other words, the heat-treatment time and temperature are determined experimentally depending on the properties of the treatment object, that is, the film to be heat-treated and the thin film formed on it.

If the retaining temperature of preheating furnace 4a is set lower than the temperature of heat-treatment furnace 4b, the scratches and defects caused by quickly heating film roll 1a can be minimized without causing problems in practical applications. It, however, depends on the retaining time, which is preferably within [less than] 2 h until all of the film on the roll-out side is fed out. If said time is longer than 2 h, the film on the roll-out side will start to undergo thermal shrinkage. As a result, many scratches will occur. There should be no problems when the retaining temperature is kept below the glass transition point of the film.

In the following, the present invention will be explained in more detail with reference to application examples.

Application Example 1

A biaxially stretched polyethylene terephthalate film with a width of 400 mm and a thickness of 100 μm was loaded into a vacuum deposition apparatus; the vacuum tank was exhausted until reaching a vacuum degree of 1×10^{-5} Torr. After that, In_2O_3/SnO_2 (SnO_2 [present at] 5 wt%) was evaporated under heating by using electron beams to form an indium/tin oxide film. Film 1 formed this way was prepared at a length of 200 m.

Spacer 2 was prepared by winding a polyethylene terephthalate film with a thickness of 125 μm and a width of 15 mm into a roll with a length of 200 m.

The roll 1a of film 1 having said indium/tin oxide film was installed on roll-out shaft 1b, while the roll 2a of spacer 2 was installed on spacer installation shaft 2b. Film 1 was fed out until reaching the winding [wind-up] shaft 3a of a winding machine (not shown in the figure). The temperature of preheating furnace 4a was set to 110°C so that continuous winding became possible.

Next, at the site where the temperature of heat-treatment furnace 4b rose to 140°C, the winding machine was rotated and heated to wind [the film] into roll 3. It took about 1 min before film 1 was wound after entering heat-treatment furnace 4b. The film was wound for

about 1 h. Within 1 min after the film entered heat-treatment furnace 4b, the surface temperature of the film reached 138°C.

After winding was finished, after a 24 h of heat-treatment performed at 140°C (in this case, rotation of the winding machine was stopped), heat-treatment furnace 4b was cooled off, and film roll 3 was removed. The two ends of the film were cut off by 25 mm by a slitter-attached winding machine to eliminate the spacers in order to eliminate the deformation and contaminated part caused by the spacers at the ends of the film.

The transparent electroconductive film obtained this way had a good surface resistance value, surface resistance linearity [uniformity], and [possibly "light"] transmission ability as well as few variations along both the length direction and width direction of the film. Also, the examination of the appearance showed a good appearance with no damage at all.

The thermal shrinkage percentages in the length direction (MD) and width direction (TD) of the film after 30 min of heating at 120°C were 0.9% and 0.8% for the used film, respectively. After the heat-treatment, the thermal shrinkage percentages became 0.1% and less than 0.1%, respectively. The thermal shrinkage percentage was within such a range at all sites along the length direction of the film.

Application Example 2

The same film 1 and spacers 2 as those used in Application Example 1 were set in preheating furnace 4a every 200 m* and fed out to winding shaft 3a. The temperature of the preheating furnace 4a was set to the air temperature of 30°C, [which is] below the glass transition temperature.

The temperature of heat treatment furnace 4b was then raised; at the site where the temperature was raised to 140°C, the winding machine was rotated and heated while the film was wound into roll 3. It took about 2 min before film 1 was wound after entering heat-treatment furnace 4b. The film was wound for about 2 h. Within 1 min after the film entered heat-treatment furnace 4b, the surface temperature of the film reached 138°C.

After the winding, the heat-treatment was carried out, with spacers 2 being removed in the same way as described in Application Example 1.

Like the film obtained in Application Example 1, the transparent electroconductive film obtained this way had a good surface resistance value, surface resistance linearity, and transmission ability as well as few variations along both the length direction and width direction of the film. Also, the examination of the appearance showed a good appearance with no damage

* [Possibly meaning "in lengths of 200 m".]

at all. The film also showed the same good thermal shrinkage percentage as that in Application Example 1.

Comparative Example

200 m of film and spacers (width: 15 mm, thickness: 125 μm, length: 200 m) prepared in the same way as described in Application Examples 1 and 2 were wound in the same way as described in Application Example 1, with the temperatures of both preheating furnace 4a and heat-treatment furnace 4b being set to 30°C. After that, the temperature of heat-treatment furnace 4b was set to 140°C, and heat-treatment was performed for 24 h.

The length of the film cut off at the two ends for eliminating the spacers was 30 mm. The [film] loss was increased.

As far as the surface resistance value after the heat-treatment is concerned, on the innermost peripheral side and the outermost peripheral side during the heat-treatment, the surface resistance value was almost the same within about 6 cm from the two ends of the film in the width direction. However, the surface resistance value in the middle excluding the two ends in the width direction and in the center of the roll was 1.5 times or higher than that of the outermost periphery. The reason is as follows. During the heat-treatment, the temperature rose slowly in the center along the length and width directions of the rolled up film. The temperature did not rise high enough in 24 h so that the heat-treatment was not fully carried out. Also, fine scratches were observed along the length direction of the film. The film had a low-quality appearance.

As far as the thermal dimensional stability is concerned, the thermal shrinkage percentages in the MD and TD directions of the film were 0.9% and 0.8%, respectively. After the heat-treatment, the thermal shrinkage percentages became 0.1% and lower than 0.1% on the outermost periphery and innermost periphery [respectively] along the length direction. The thermal shrinkage percentages in the center, however, were 0.4% and 0.4%, respectively. This indicated that the heat-treatment was not fully carried out.

The present invention has been explained in detail based on application examples. The present invention, however, is not limited to these application examples.

Brief description of the figures

Figure 1 is a diagram explaining the heat-treatment apparatus used in the application example of the present invention. Figure 2 is a cross-sectional view explaining the film roll on the roll-out side in Figure 1.

1 Film

2 Spacer

4a Preheating furnace

4b Heat-treatment furnace

Patent Application	Teijin Ltd.
Agent	Sumihara Maeda

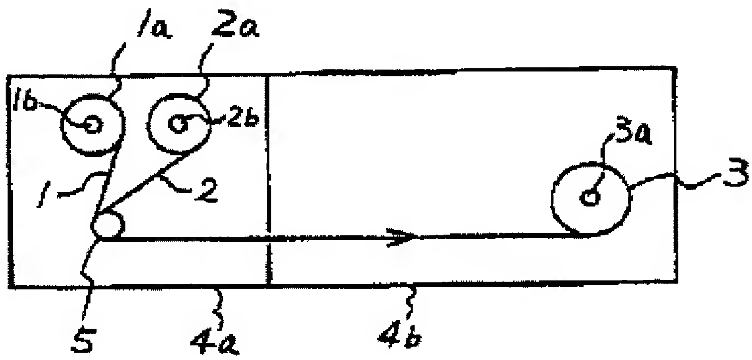

Figure 1

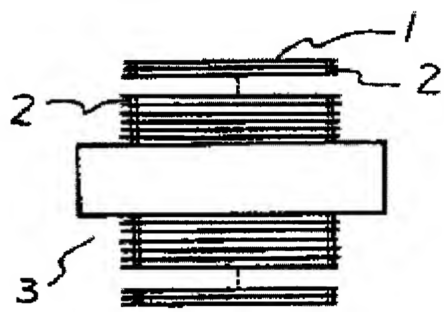

Figure 2